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IS 2012 (2006): Red Phosphorus -Specification [CHD 26: Explosives and Pyrotechnics]

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भारतीय मानक

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(पहला पुनरीक्षण)

Indian Standard

RED PHOSPHORUS — SPECIFICATION

(First Revision)

ICS 71.060.01

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 2006 **Price Group 3**

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Explosives and Pyrotechnic Sectional Committee had been approved by the Chemical Division Council.

Red phosphorus is one of the most important raw materials used in the manufacture of safety matches. It is also used in appreciable quantities in metallurgy and for the manufacture of fireworks. Though the present indigenous requirements for this important material are met by imports, efforts are being made to start its manufacture in India.

This standard was published in 1961 to cover the technical provisions relating to the material without including all the necessary provisions of a contract. In the preparation of this standard, assistance was derived from the company standards of Swedish Match Co, and Hooker Chemical Co (USA) and the US Joint Army-Navy Specification (Army : 4-503-300C; Navy : 51P37) on this subject.

In this revision, requirement of red phosphorus has been reduced from 99.0 to 97.5 percent by mass, minimum and further material shall contain no white phosphorus and requirement of increase in acidity on exposure to moisture for 21 days has been deleted. Atomic absorption spectrophotometric methods for determination of copper and iron as indicated in IS 11123 : 1984 and IS 13320 : 1992 have been incorporated.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

RED PHOSPHORUS — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for red phosphorus, suitable for match industry and general industrial use.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
264 : 2005	Nitric acid (*third revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
266 : 1993	Sulphuric acid (*third revision*)
323 : 1959	Specification for rectified spirit (*revised*)
1070 : 1992	Reagent grade water (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (*first revision*)

3 REQUIREMENTS

3.1 The material shall be in the form of dark red amorphous powder or granules consisting essentially of phosphorus. It shall be free from visible contamination.

3.2 The material, when tested by the methods prescribed in Annex A, shall comply with the requirements given in Table 1. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

Table 1 Requirements for Red Phosphorus

(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Free moisture, percent by mass, *Max*	0.2	A-2
ii)	Matter soluble in water, percent by mass, *Max*	1.5	A-3
iii)	Red phosphorus, percent by mass, *Min*	97.5	A-4
iv)	Acidity (as H_3PO_4), percent by mass, *Max*	0.1	A-6

3.3 White Phosphorus Content

The material shall be found to contain no white-phosphorus when tested by the method prescribed in A-5.

3.4 Additional Requirements for Defence Purposes

The material for defence purposes shall, in addition to the requirements given in Table 1, comply with the following requirements when tested as prescribed in A-7.

a) Copper (as Cu), parts per million, Maximum-50

b) Iron (as Fe), parts per million, Maximum-50

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in air-tight containers. Unless otherwise agreed to between the purchaser and the vendor, containers shall have a nominal capacity of 5 kg and 50 kg, the 5 kg containers being tin-plated and 50 kg containers being lined with an adequate polyethylene liner (not less than 0.1 mm thick).

4.2 Marking

Each container shall be legibly marked with the following information:

a) Name of the material;

b) Name of the manufacturer and recognized trade-mark, if any;

c) Net mass of the material in the container;

d) Month and year of manufacture; and

e) Caution label 'Flammable' with the appropriate symbol for labelling dangerous goods [*see* IS 1260 (Part 1)].

4.2.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

4.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

The methodology for the drawal of representative samples and criteria for conformity with this standard shall be as prescribed in Annex B.

ANNEX A

(*Clauses* 3.2, 3.3, 3.4 and *Table* 1)

ANALYSIS OF RED PHOSPHOROUS

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS1070) shall be used in tests.

A-2 DETERMINATION OF MOISTURE

A-2.1 Apparatus

A-2.1.1 *Flat Bottomed Glass Dish*, approximately 60 mm diameter and 30 mm height with ground glass lid.

A-2.1.2 *Vacuum Desiccator*, with phosphorus pentoxide as desiccant.

A-2.2 Procedure

A-2.2.1 Heat the dish with lid in the oven for about 30 min. Cool it and weigh, weigh accurately about 5 g of the sample, replace the lid and weigh accurately.

Uncover the dish and place it in a vacuum desiccator (10 mm or less of mercury) over phosphorus pentoxide for three hours. Weigh it with lid.

A-2.3 Calculation

$$\text{Moisture, percent by mass} = \frac{(M_2 - M_3)\times 100}{(M_2 - M_1)}$$

where

M_1 = mass of the empty dish, in g;
M_2 = mass of the material and the dish, in g; and
M_3 = mass of the material and the dish after drying, in g.

A-3 DETERMINATION OF MATTER SOLUBLE IN WATER

A-3.1 Procedure

Weigh accurately about 5 g of sample in 500 ml beaker. Add 100 ml of distilled water. Stir well. Allow to stand for 4 h at room temperature with occasional stirring. Filter through Whatman No. 2 filter paper, collect the filtrate and make up to 250 ml in a volumetric flask. Pipette out 50 ml of filtrate into a tared 150 ml beaker. Evaporate the solution to 5 ml on a hot plate and finally dry the residue to constant mass in an oven at 100°C.

A-3.2 Calculation

$$\text{Matter soluble in water, percent by mass} = \frac{(M_1 - M_2)\times 500}{M_1}$$

where

M_1 = mass of the material taken for the test, in g; and
M_2 = mass of the residue left after extraction, in g.

A-4 DETERMINATION OF RED PHOSPHOROUS

A-4.1 Reagents

A-4.1.1 *Iodine*

A-4.1.2 *Concentrated Nitric Acid* — *See* IS 264.

A-4.1.3 *Ammonium Citrate*

A-4.1.4 *Dilute Ammonium Hydroxide* — 1: 4 (*v*/*v*).

A-4.1.5 *Dilute Hydrochloric Acid* — Approximately 5 N.

A-4.1.6 *Magnesia Mixture* — Dissolve 110 g of magnesium chloride ($MgCl_2.6\,H_2O$) in a small amount of water. Add 288 g of ammonium chloride and 700 ml of ammonium hydroxide (sp gr 0.90). Dilute the solution to 2 litres with water. Allow to stand for several hour, filter and transfer the solution to a 2 litre glass-stoppered bottle.

NOTE — As the reagent becomes old, it will be necessary to filter off the silica, that is, gradually accumulated from the reagent bottle.

A-4.1.7 *Ammonium Hydroxide* — Sp gr 0.90.

A-4.1.8 *Ammonium Nitrate Solution* — Saturated.

A-4.2 Procedure

A-4.2.1 *Prepared Solution*

Weigh accurately about 1g of the material into a 300 ml conical flask, add 4 to 5 ml of water and mix well. Add in small portions 12 g of iodine to dissolve the material. Then gently heat the flask and add carefully 50 ml of concentrated nitric acid. After the dissolution of the phosphorus iodide, add 20 ml more of concentrated nitric acid and boil gently until all iodine has been expelled. Dilute, filter into a 500 ml measuring flask and add water to the mark. This shall be the prepared solution.

A-4.2.2 Take 5 g of ammonium citrate and 25 ml of water in a 200 ml beaker and add 25 ml of the prepared solution (*see* **A-4.2.1**). Make the solution alkaline with dilute ammonium hydroxide and then acidic with dilute hydrochloric acid. Add dropwise 25 ml of cold magnesia mixture with constant stirring. Discontinue the stirring when the solution becomes cloudy and allow the precipitate to settle for about 10 min. Add 20 ml of ammonium hydroxide again with constant stirring. Leave the precipitate to settle for 4 h or, preferably overnight.

A-4.2.3 Filter through quantitative filter paper and wash the precipitate several times with small quantities of dilute ammonium hydroxide. Dissolve the precipitate in dilute hydrochloric acid and dilute it approximately to 50 ml in a 200 ml beaker. Again precipitate the magnesium ammonium phosphate according to the procedure given in **A-4.2.2**. Filter the precipitate through an ashless filter paper and wash the precipitate with dilute ammonium hydroxide until free of chloride. Place the filter paper with the precipitate in a tared porcelain crucible, add a few drops of ammonium nitrate solution, and heat the crucible over a low flame until the paper is charred. Then place the crucible in a muffle furnace at 1 100°C for 1 h, cool in a desiccator and weigh the residue as a magnesium pyrophosphate. Repeat the ignition until a constant weight is obtained.

A-4.3 Calculation

Red phosphorous (as P),

$$\text{percent by weight} = \frac{556.8 \times M_2}{M_1}$$

where

M_2 = mass of the material taken for the test, in g; and

M_1 = mass of the residue, in g.

A-5 DETERMINATION OF WHITE PHOSPHOROUS

A-5.1 Procedure

Take 10 g of the sample in a stoppered measuring cylinder; add 20 ml of carbon disulphide. Shake well. Allow it to settle for 2 h. Place a few drops of the clear supernatant liquid upon a piece of the copper sulphate paper (made by soaking filter paper in 20 percent copper sulphate solution and drying in the oven at 60°C). Absence of any brown stain indicates the absence of white phosphorus.

A-6 DETERMINATIION OF ACIDITY

A-6.1 Reagents

A-6.1.1 *Phenolphthalein Indicator* — Dissolve 0.1 g of phenolphthalein in 100 ml of rectified spirit (*see* IS 323).

A-6.1.2 *Sodium Chloride* — Neutral.

A-6.1.3 *Standard Sodium Hydroxide Solution* — 0.1 N.

A-6.2 Procedure

Weigh accurately about 5.0 g of the material and transfer it to 250 ml conical flask. Add 100 ml of water. Heat the mixture to boiling and filter through a Buckner funnel with gentle suction. Wash the residue several times with water. Add a few drops of phenolphthalein indicator and about 10 to 15 g of sodium chloride. Cool the solution in an ice bath and titrate with standard sodium hydroxide solution.

A-6.3 Calculation

$$\text{Acidity (as } H_3PO_4\text{), percent by mass} = \frac{V \times N}{M} \times 4.9$$

where

V = volume of standard sodium hydroxide solution used for the titration, in ml;

N = normality of standard sodium hydroxide solution; and

M = mass of the material taken for the test, in g.

A-7 DETERMINATION OF COPPER AND IRON

A-7.1 Apparatus

A-7.1.1 *Atomic Absorption Spectrophotometer*

NOTE — The manufacturer's instructions shall be followed for all instrumental analysis.

A-7.2 Reagents

A-7.2.1 *Concentrated Nitric Acid* — *See* IS 264.

A-7.2.2 *Concentrated Sulphuric Acid* — *See* IS 266.

A-7.2.3 *Hydrochloric Acid* — Approximately 6 N, *see* IS 265.

A-7.2.4 *Standard Copper Solution* — Dissolve 1 g of 99.9 percent, minimum pure copper granules in 50 ml of 1:1 nitric acid. Boil to expel nitrous fumes and cool. Make up the solution to 1 000 ml with water. Each ml of solution contains 1 000 ppm of copper. Make appropriate dilution to prepare standard solutions of required concentration.

A-7.2.5 *Standard Iron Solution* — Dissolve 1 g of 99.9 percent minimum pure iron granules in 50 ml of 1:1 nitric acid. Boil to expel nitrous fumes and cool. Make up the solution to 1 000 ml with water. Each ml of solution contains 1 000 ppm of iron. Make approximate dilution for preparing standard solutions of required concentration.

A-7.3 Procedure

Weigh accurately about 1 g of material in a 250 ml beaker. Add 50 ml of water. Add 10 ml of concentrated HNO_3 and rapidly boil down to 3 ml on a hot plate. At this stage, rapid evolution of brown nitrous fumes occurs. Remove the beaker from the hot plate and add 5 ml of concentrated sulphuric acid. Heat to fumes and cautiously evaporate to near dryness. Cool and dissolve it in 6N hydrochloric acid and dilute to 100 ml in a volumetric flask with 6N hydrochloric acid.

A-7.3.1 Determine the copper and iron content by atomic absorption spectrophotometer using wave length 324.8 nm for copper and 248.3 nm for iron.

The element content, expressed as ppm is given by the formula:

$$\frac{C \times V}{M}$$

where

C = concentration of the element in test solution, in mg/litre;

V = volume, in ml; and

M = mass of the test portion, in g.

ANNEX B

(*Clause* 5)

SAMPLING OF RED PHOSPHORUS

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B.1.1 Samples shall be taken in a protected place.

B-1.2 For sampling, an aluminium sampling thief shall be used. It shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from an adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for, sampling shall be mixed as thoroughly as possible, by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed airtight with a stopper after filling, and marked with full details of the materials and the date of sampling.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from normal atmospheric temperature.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers of the same size in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from the lot shall depend on the size of the lot as given in Table 2.

Table 2 Number of Containers to be Selected for Sampling

(*Clause* B-2.2)

Sl No.	Lot Size	Number of Containers to be Chosen	
	Metric tonne	50-kg Drums	5-kg Tins
(1)	(2)	(3)	(4)
i)	Below 2	2	3
ii)	2-3	3	4
iii)	3-4	3	5
iv)	4-5	4	6
v)	5-6	4	6
vi)	6-7	4	6
vii)	Over 7	5	7

B-2.3 These containers shall be chosen at random from the lot and in order to ensure the randomness of selection, a random number table as agreed to between the purchaser and the vendor shall be used. In case such a table is not available, the following procedure shall be adopted.

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as

1, 2, 3.......etc, up to *r* and so on, where $r = N/n$, *N* being the total number of containers in the lot and *n* the number of containers to be chosen (*see* Table 2). In case *r* comes out to be a fractional number, its value shall be taken to be equal to the integral part of it. Every *r*th container thus counted shall be withdrawn from the lot to give a sample for test.

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Sample

B-3.1.1 Draw with the sampling thief a small portion of the material from different parts of each container selected for sampling. The total quantity of the material drawn form each container shall be sufficient to conduct the test for all the characteristics given in 3 and shall not exceed 1 kg.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken out from each selected container and well mixed up together so as to form a composite sample weighing not less than 1 kg. This composite sample shall be divided into three equal parts; one for the purchaser, another for the vendor and the third to be used as referee sample.

B-3.1.3 The remaining portion of the material from each container (after a small quantity needed for formation of composite sample has been taken out) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed airtight with stoppers and labelled with all the particulars of sampling given in **B-1.7**. The material in each such sealed bottle shall constitute a test sample. These individual samples shall be separated into three identical sets of test samples in such a way that each set has a test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the vendor and the third shall be used a referee sample.

B-3.2 Referee Sample

The referee sample shall consist of the composite sample (*see* **B-3.1.2**) and a set of test samples (*see* **B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the vendor. These shall be kept at a place agreed to between the purchaser and the vendor and shall be used in case of a dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of acidity shall be conducted individually on each of the samples constituting a set of test samples (*see* **B-3.1.3**).

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample (*see* **B-3.1.2**).

B-5 CRITERION FOR CONFORMITY

B-5.1 For Individual Sample

From the various test results for acidity, calculate the following values:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of test results}}{\text{Number of test results}}$$

Range (R) = The difference between the maximum and the minimum test results.

B-5.1.1 The material shall be taken to have satisfied the requirement specified if

$(\bar{X} + 0.6R)$ does not exceed 0.1.

B-5.2 For Composite Sample

For the remaining characteristics prescribed in Table 1 the test results on the composite sample shall meet the corresponding requirements specified in col 4 of Table 1.

B-5.3 A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics listed in Table 1.

B-5.4 If the requirement for any of the characteristics is not met, the lot shall be declared to have not satisfied the requirements of the specification.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Explosives and Pyrotechnics Sectional Committee, CHD 26

Organization	*Representative(s)*
Department of Explosives, Nagpur	Shri M. Anbunathan (***Chairman***) Shri K. N. Ghosh (*Alternate*)
A. J. Research Centre, Sivakasi	Shri A. Ramamurthy Shri P. Kanagaraj (*Alternate*)
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Member Secretary
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